

NC DEQ/DWR LABORATORY CERTIFICATION BRANCH

LABORATORY NAME:		CERT #:	
PRIMARY ANALYST:		DATE:	
NAME OF PERSON COMPLETING CHECKLIST (PRINT):			
SIGNATURE OF PERSON COMPLETING CHECKLIST:			

Parameter: **Cyanide**
Method: **Standard Method 4500-CN⁻ E – 2016 (Aqueous)**
Technology: **Spectrophotometric, Manual**

EQUIPMENT:

Spectrophotometer, for use at 578 nm, providing a light path of 10 mm or longer. Model:	Boiling flask, 1 L, with inlet tube and provision for water-cooled condenser.
Filter photometer, providing a light path of at least 10 mm and equipped with a red filter having maximum transmittance at 570 to 580 nm. Model:	Gas absorber, with gas dispersion tube equipped with medium-porosity fritted outlet.
Heating element, adjustable.	Ground glass ST joints, TFE-sleeved or with an appropriate lubricant for the boiling flask and condenser. Neoprene stopper and plastic threaded joints also may be used.

DISTILLATION REAGENTS:

<i>Sodium hydroxide solution:</i> Dissolve 40 g NaOH in water and dilute to 1 L.
<i>Magnesium chloride reagent (optional):</i> Dissolve 510 g MgCl ₂ · 6H ₂ O in water and dilute to 1 L.
<i>Sulfuric acid, H₂SO₄, 1 + 1.</i>

Lead carbonate, PbCO ₃ , powdered.
Sulfamic acid, NH ₂ SO ₃ H.

ANALYSIS REAGENTS AND STANDARDS

<i>Chloramine-T solution:</i> Dissolve 1.0 g white powder in 100 mL water. Prepare fresh daily.
<i>Stock cyanide solution:</i> Dissolve approximately 1.6 g NaOH and 2.51 g KCN in 1 L reagent water. (CAUTION- KCN is highly toxic; avoid contact or inhalation.) Standardize against standard silver nitrate (AgNO ₃) titrant as described in 4500-CN ⁻ .D.4, using 25 mL KCN solution. Check titer weekly because the solution gradually loses strength; 1 mL = 1 mg CN ⁻ .
<i>Standard cyanide solution:</i> Based on the concentration determined for the KCN stock solution calculate volume required (approximately 10 mL) to prepare 1 L of a 10 µg CN ⁻ /mL solution. Dilute with the NaOH dilution solution. Dilute 10 mL of the 10 µg CN ⁻ /mL solution to 100 mL with the NaOH dilution solution; 1.0 mL = 1.0 µg CN ⁻ . Prepare fresh daily and keep in a glass-stoppered bottle. (CAUTION- Toxic; take care to avoid ingestion.)

<i>Pyridine-barbituric acid reagent:</i> Place 15 g barbituric acid in a 250-mL volumetric flask and add just enough water to wash sides of flask and wet barbituric acid. Add 75 mL pyridine and mix. Add 15 mL conc hydrochloric acid (HCl), mix, and cool to room temperature. Dilute to volume and mix until barbituric acid is dissolved. The solution is stable for approximately 6 months if stored in an amber bottle under refrigeration; discard if precipitate develops.
<i>Acetate buffer:</i> Dissolve 410 g sodium acetate trihydrate, NaC ₂ H ₃ O ₂ · 3H ₂ O, in 500 mL of water. Add glacial acetic acid to adjust to pH 4.5 S.U., approximately 500 mL.
<i>Sodium hydroxide dilution solution:</i> Dissolve 1.6 g NaOH in 1 L reagent water.

PLEASE COMPLETE CHECKLIST IN INDELIBLE INK

Please mark Y, N or NA in the column labeled LAB to indicate the common lab practice and in the column labeled SOP to indicate whether it is addressed in the SOP.

	GENERAL	LAB	SOP	EXPLANATION
1	Is the SOP reviewed at least every 2 years? What is the most recent review/revision date of the SOP? [15A NCAC 2H .0805 (a) (7)] ANSWER:			Quality assurance, quality control, and Standard Operating Procedure documentation shall indicate the effective date of the document and be reviewed every two years and updated if changes in procedures are made. Verify proper method reference. During review notate deviations from the approved method and SOP.
2	Are all revision dates and actions tracked and documented? [15A NCAC 2H .0805 (a) (7)]			Each laboratory shall have a formal process to track and document review dates and any revisions made in all quality assurance, quality control and SOP documents.

3	Is there North Carolina data available for review?			If not, review PT data
	PRESERVATION and STORAGE	L A B	S O P	EXPLANATION
4	Are samples tested and treated for known interferences at the time of collection and prior to pH adjustment and thermal preservation? [ASTM D7365–09a (15), Section 8.3.5]			Treat the sample immediately upon sample collection using any or all of the following techniques as necessary, followed by adjustment of the sample to pH > 10 S.U. and refrigeration.
5	Are samples checked for oxidizers (e.g., chlorine) and other known interferences at the time of collection? [40 CFR 136.3 Table II] [ASTM D7365–09a (15), Section 8.3.9]			<p>40 CFR 136.3 Table II footnote 5: ASTM D7365–09a specifies treatment options for samples containing oxidants (e.g., chlorine) for cyanide analyses.</p> <p>NC WW/GW LCB Policy requires documentation that demonstrates oxidizers are <0.5 mg/L. TRC strips or DPD powder may be used.</p> <p>Footnote 6: Sampling, preservation and mitigating interferences in water samples for analysis of cyanide are described in ASTM D7365–09a (15). There may be interferences that are not mitigated by the analytical test methods or D7365–09a (15). Any technique for removal or suppression of interference may be employed, provided the laboratory demonstrates that it more accurately measures cyanide through quality control measures described in the analytical test method. Any removal or suppression technique not described in D7365–09a (15) or the analytical test method must be documented along with supporting data.</p>
6	If known or suspected to be present, how is chlorine detected and mitigated? [ASTM D7365 – 09a (15), Sections 8.3.9 and 8.3.9.1]			<p>8.3.9 <i>Chlorine, Hypochlorite, or other Oxidant</i>—Add a reducing agent only if an oxidant (for example, chlorine) is known or suspected to be present. Samples can be screened for oxidants by placing a drop of sample on a potassium-iodide (KI) starch paper. Reducing agents shown to be effective at removing oxidants are sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3$), ascorbic acid, sodium arsenite (NaAsO_2), or sodium borohydride (NaBH_4). However, some of these reagents have shown to produce a positive or negative cyanide bias, depending on other substances in the sample and the analytical method used. Warning— When NaBH_4 is added to samples that contain arsenic, the formation of toxic arsine gas can occur. NaBH_4 can also produce hydrogen gas which could result in an explosion upon acidification of the sample.</p> <p>8.3.9.1 Unless the method specifies otherwise, sodium arsenite is the preferred reducing agent. Ascorbic acid can also be used; however, analysis must be performed within 24 hours to avoid cyanide degradation unless the holding time has been determined as described in Practice D4841 and Section 8.3.2. Methods recommending ascorbic acid specify adding 0.1 to 0.6 g/L. If NaAsO_2 is used, 100 mg/L NaAsO_2 will reduce more than 50 mg/L chlorine. After adding reducing agent, test the sample with a KI test strip to make sure all</p>

			<p>oxidant is removed. If oxidant remains, add more reducing agent, but avoid adding excess. Whatever agent is used, it should be tested to ensure that cyanide results are not affected adversely. Whenever a reducing agent is used to dechlorinate, nitrite could possibly form; therefore, sulfamic acid should be added during the distillation step for total cyanide determinations (for example, Test Methods D7284 and D2036 Test Method A) as described in Section 8.4.5.</p> <p>Ascorbic acid is no longer a recommended preservative for cyanide samples because it functions as a carbon donor in the presence of nitrite or nitrate, generating cyanide during distillation. Sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3$) is an adequate dechlorinating agent as long as it is not used in excess. Sodium arsenite (NaAsO_2) also may be used, but it is a hazardous material.</p>
7	<p>If known or suspected to be present, how is sulfide detected and mitigated? [SM 4500-CN- B-2016 (2)] [SM 4500-CN- B-2016 (3) (b)] [ASTM D7365-09a (15), Section 8.3.6.2]</p>		<p>Oxidized products of sulfide convert cyanide to thiocyanate rapidly, especially at elevated pH. Sulfides also interfere with several cyanide analysis methods. If sulfides are suspected, test for S^{2-} by placing a drop of sample on lead acetate test paper previously moistened with acetic acid buffer solution at pH 4 S.U. (Section 4500-Cl.C.3e). If the paper darkens, then S^{2-} is present. Test suspect samples as soon as possible to avoid cyanide loss, and promptly dilute sulfide-positive samples with reagent water (to mitigate sulfide's effect). Avoid excessive dilution, which would raise the cyanide reporting limit above a regulatory limit.</p> <p>Some analytical methods specify the use of lead carbonate or lead acetate to precipitate sulfide; however, sulfide and cyanide can form thiocyanate in the presence of lead sulfide causing decreased cyanide recoveries; therefore, lead carbonate and lead acetate should be avoided unless there is no other means to mitigate the sulfide or if the sample cannot be diluted as described in 8.3.6.1</p> <p>If the sample cannot be diluted, sulfide is removed by treating the sample with small increments of powdered lead carbonate or with the dropwise addition of lead acetate solution. Black lead sulfide precipitates in samples containing sulfide. Repeat the operation until no more lead sulfide forms, as indicated by testing the supernatant liquid with lead acetate test paper as indicated in 8.3.6. Immediately (within 15 minutes) filter with coarse filter paper (5 μm), then stabilize the sample according to 8.3.5. If the sample contains colloids that may contribute to the total cyanide concentration, filter the sample prior to adding the lead, then recombine the solids with the filtrate prior to analysis. Samples that are known or suspected to contain sulfides should be analyzed as soon as possible to avoid cyanide degradation. Treat 25 mL more than required for the distillation to provide sufficient filtrate volume.</p>

8	After mitigation of interferences, are samples preserved within 15 minutes of collection with NaOH to pH of > 10 S.U.? [40 CFR 136.3 Table II]			Preservation not required if analyzed within 15 minutes. However, mitigation of interferences would still be required.
9	Are samples iced to above freezing but ≤ 6 °C during shipment? [40 CFR 136.3 Table II and footnote 18]			40 CFR footnote 2 allows 15 minutes for sample preservation, including thermal. This means that if a sample is received in the lab within 15 minutes it is not required to be on ice. Document temperature downward trend for short transport samples.
10	Upon receipt at the laboratory, are samples tested for pH, sulfides and oxidants to verify proper mitigation of interferences and subsequent preservation? [ASTM D7365–09a (15), Section 8.4.1]			Upon receipt of sample(s) at the laboratory, verify that interferences are not present as indicated in this practice. At a minimum, test unknown samples for pH, sulfides and oxidants using pH indicator test strips, lead acetate test strips <u>previously moistened with acetate buffer</u> and KI starch paper, respectively. Document and mitigate any interference discovered in the laboratory, and if necessary, recollect the sample to mitigate the interference at time of collection. If resampling is not possible, qualify the result by describing the potential interference.
11	Are samples stored at ≤ 6°C, without freezing? [40 CFR 136.3 Table II and footnote 18]			
12	Are samples analyzed within 14 days of collection? [40 CFR 136.3 Table II]			
13	Are samples analyzed within 24 hours when ascorbic acid is used as the reducing agent? [ASTM D7365–09a (15), Section 8.3.9.1]			Note: If Ascorbic acid is used, analysis must be performed within 24 hours to avoid cyanide degradation unless the holding time has been determined as described in Practice D4841 and Section 8.3.2.
	PROCEDURE – Distillation	L A B	S O P	EXPLANATION
14	Is a 500 mL sample, containing not more than 10 mg CN ⁻ /L, used for the distillation? [SM 4500-CN ⁻ C–2016 (4)(a)]			Add 500 mL sample, containing not more than 10 mg CN ⁻ /L (diluted if necessary with reagent water) to the boiling flask. If a higher CN ⁻ content is anticipated, use the spot test (4500-CN ⁻ . K) to approximate the required dilution. Other equivalent apparatus, including scaled-down apparatus, may be used if the scaled-down apparatus volumes preserve the same ratios of sample volume to digestion reagents volume as specified in the procedure (4500-CN ⁻ .C.4).
15	Is at least 10 mL of NaOH solution added to the absorber tube? [SM 4500-CN ⁻ C–2016 (4)(a)]			Add 10 mL NaOH solution to the gas scrubber and dilute, if necessary, with reagent water to obtain an adequate liquid depth in the absorber. Do not use more than 225 mL total volume of absorber solution.
16	Is lead carbonate or lead acetate used to precipitate sulfide? [SM 4500-CN ⁻ C–2016 (4)(a)] [ASTM D7365–09a (15), Section 8.3.6.2]			SM says, “When S ²⁻ generation from the distilling flask is anticipated add 50 or more mg powdered PbCO ₃ to the absorber solution to precipitate S ²⁻ .” However, the ASTM method says “Some analytical methods specify the use of lead carbonate or lead acetate to precipitate sulfide; however, sulfide and cyanide can form thiocyanate in the presence of lead sulfide causing decreased cyanide recoveries; therefore, lead carbonate and lead acetate should be avoided unless there is no other means to mitigate the

				sulfide”.
17	Is sufficient air flow pulled through the apparatus to prevent backup in the delivery tube and not raise the liquid level in the absorber tube by more than 10mm? [SM 4500-CN ⁻ C-2016 (4)(a)]			Adjust suction so at least 1 air bubble/s enters the boiling flask. This air rate will carry HCN gas from flask to absorber and usually will prevent a reverse flow of HCN through the air inlet. If this air rate does not prevent sample backup in the delivery tube, increase air-flow rate to 2 air bubbles/s. Observe air purge rate in the absorber where the liquid level should be raised not more than 6.5 - 10 mm. Maintain air flow throughout the reaction.
18	Is 2 g sulfamic acid added through the air inlet tube and wash down with reagent water? [4500-CN ⁻ C-2016 (4)(b)]			Add 2 g sulfamic acid through the air inlet tube and wash down with reagent water.
19	Is 50 mL 1+1 H ₂ SO ₄ and 20 mL MgCl ₂ reagent added through the air inlet tube and rinsed down with reagent water? [SM 4500-CN ⁻ C-2016 (4)(c)]			Add 50 mL 1 + 1 H ₂ SO ₄ through the air inlet tube. Rinse tube with reagent water and let air mix flask contents for 3 min. Optionally , add 20 mL MgCl ₂ reagent through air inlet and wash down with stream of water. A precipitate that may form re-dissolves on heating.
20	Is the sample allowed to air mix for 3 minutes prior to applying heat? [SM 4500-CN ⁻ C-2016 (4)(c)]			Rinse tube with reagent water and let air mix flask contents for 3 min.
21	Is the sample brought to a rapid boil without allowing the condenser inlet to flood or permitting vapors to rise more than halfway into condenser? [SM 4500-CN ⁻ C-2016 (4)(d)]			Heat with rapid boiling, but do not flood condenser inlet or permit vapors to rise more than halfway into condenser.
22	Is the sample allowed to reflux for at least 1 hour? [SM 4500-CN ⁻ C-2016 (4)(d)]			Adequate refluxing is indicated by a reflux rate of 40 to 50 drops/min from the condenser lip. Reflux for at least 1 h.
23	Is the heat removed and the sample allowed to cool with continued air flow for at least 15 minutes? [SM 4500-CN ⁻ C-2016 (4)(d)]			Discontinue heating but continue air flow for 15 min.
24	Once cooled, is the absorption solution quantitatively transferred to a 250-mL volumetric flask and brought to volume with reagent water? [SM 4500-CN ⁻ C-2016 (4)(d)]			Cool and quantitatively transfer absorption solution to a 250-mL volumetric flask. Rinse absorber and its connecting tubing sparingly with reagent water and add to flask. Dilute to volume with reagent water and mix thoroughly.
	PROCEDURE – Sample Analysis	L A B	S O P	EXPLANATION
25	Is a portion of absorption solution added to a 50-mL volumetric flask and diluted to 40 mL with NaOH dilution solution? [SM 4500-CN ⁻ E-2016 (4)(b)]			Pipet a portion of absorption solution into a 50-mL volumetric flask and dilute to 40 mL with NaOH dilution solution.
26	Is 1 mL acetate buffer and 2 mL chloramine-T solution added to the flask and mixed with the sample by inverting twice? [SM 4500-CN ⁻ E-2016 (4)(b)]			Add 1 mL acetate buffer and 2 mL chloramine-T solution, stopper, and mix by inversion twice.
27	Is this mixture allowed to stand for exactly 2 minutes before adding 5 mL pyridine-barbituric acid reagent and diluting to volume with reagent water? [SM 4500-CN ⁻ E-2016 (4)(b)]			Let stand exactly 2 min . Add 5 mL pyridine-barbituric acid reagent, dilute to volume with reagent water, mix thoroughly, and let stand exactly 8 min .
28	Is the sample then allowed to stand for exactly 8 minutes before reading on the spectrophotometer? [SM 4500-CN ⁻ E-2016 (4)(b)]			See explanation above.
29	Is this instrument zeroed with reagent water at a wavelength of 578 nm? [SM 4500-CN ⁻ E-2016 (4)(b)]			Measure absorbance against reagent water at 578 nm.
30	Are all standards, samples and blanks analyzed in 10-mm cells? [SM 4500-CN ⁻ E-2016 (4)(a)]			Develop and measure absorbance in 10-mm cells as described in ¶ b below for both standards and blank.

31	Are samples with concentrations lower than 0.02 mg/L analyzed in 100-mm cells? [SM 4500-CN E-2016 (4)(a)]			For concentrations lower than 0.02 mg CN/L use 100-mm cells.
	QUALITY ASSURANCE	L A B	S O P	EXPLANATION
32	What is the concentration of each calibration curve standard? [SM 4500-CN E-2016 (4)(a)] [15A NCAC 2H .0805 (a) (7) (H) (v)] ANSWER:			For colorimetric analyses, a series of five or more non-zero standards for a curve prepared every 12 months or three or more non-zero standards for curves established each day, or standards as set forth in the analytical procedure, shall be analyzed to establish a calibration curve. A manufacturer's factory-set calibration (internal curve) shall be verified with the same number of standards and frequency as a prepared curve.
33	Does the calibration curve have a minimum correlation coefficient value of at least 0.995? [NC WW/GW LCB Policy] ANSWER:			When linear regression is used, use the minimum correlation coefficient specified in the method. If the minimum correlation coefficient is not specified, then a minimum value of 0.995 (or a coefficient of determination, r^2 , of 0.99) is required.
34	Is each calibration point back-calculated against the curve? [SM 4020 B-2014 (1) (b)]			Compare each calibration point to the curve and recalculate its concentration.
35	What is the acceptance criterion for back calculation? [SM 4020 B-2014 (1) (b)] ANSWER:			Unless otherwise specified in individual methods, if any recalculated values are not within the method's acceptance criteria—up to twice the MRL \pm 50%; between 3 and 5 times the MRL \pm 20%; or greater than 5 times the MRL \pm 10%—identify the source of any outlier(s) and correct before sample quantitation.
36	Is the calibration verified by analyzing a second-source standard at a concentration near the mid-point of the calibration curve? [SM 4020 B-2014 (1) (b)] List value of standard used. ANSWER:			Verify the initial calibration by analyzing a standard prepared from a different stock standard than that used to create the calibration curve; its concentration should be near the midpoint of the calibration range.
37	What is the acceptance criterion for the ICV? [SM 4020 B-2014 (1) (b)] ANSWER:			The analytical results for this second-source midrange standard must be within 10% of its true value.
38	What corrective action is taken if the second source ICV standard recovery is outside of established control limits? [SM 4020 B-2014 (1) (b)] [15A NCAC 2H .0805 (a) (7) (B)] ANSWER:			SM: If not, determine the cause of the error, take corrective action, and reverify the calibration. If the reverification passes, continue the analyses; otherwise, repeat the initial calibration. Rule: If quality control results fall outside established limits or show an analytical problem, the laboratory shall identify the Root Cause of the failure. The problem shall be resolved through corrective action, the corrective action process documented, and any samples involved shall be reanalyzed, if possible.
39	What is the laboratory's reporting limit? [15A NCAC 2H .0805 (a) (7) (H)] ANSWER:			For analytical procedures requiring analysis of a series of standards, the concentrations of these standards shall bracket the range of the sample concentrations measured. One of the standards shall have a concentration equal to or less than the laboratory's lowest reporting concentration for the parameter involved. All data sets shall reference the corresponding

			calibration.
40	Is a lower reporting limit standard analyzed or back-calculated with each analysis? [15A NCAC 2H .0805 (a) (7) (H)]		Laboratories shall analyze or back-calculate a standard at the same concentration as the lowest reporting concentration each day samples are analyzed.
41	What is the acceptance criterion of the lower reporting limit standard? [15A NCAC 2H .0805 (a) (7) (A)] ANSWER:		Establish laboratory control limits. Unless specified by the method or this Rule, each laboratory shall establish performance acceptance criteria for all quality control analyses.
42	What corrective action is taken if the lower reporting limit standard recovery is outside of established control limits? [15A NCAC 2H .0805 (a) (7) (B)] ANSWER:		If quality control results fall outside established limits or show an analytical problem, the laboratory shall identify the Root Cause of the failure. The problem shall be resolved through corrective action, the corrective action process documented, and any samples involved shall be reanalyzed, if possible.
43	Is a method blank analyzed with each batch of 20 or fewer samples? [SM 4020 B-2014 (5)]		Include at least one MB daily or with each batch of 20 or fewer samples, whichever is more frequent. The method blank must be exposed to all the same preservation and interference mitigation chemicals as the samples.
44	Is the NaOH dilution solution used as the calibration blank? [SM 4500-CN- E-2016 (4)(a)]		Measure absorbance of blank (0.0 mg CN ⁻ /L) using 40 mL NaOH dilution solution and procedures for color development.
45	Is the blank concentration $\leq \frac{1}{2}$ the concentration of the lowest reporting limit? [15A NCAC 2H .0805 (a) (7) (H) (i)]		Rule: The concentration of reagent, method, and calibration blanks shall not exceed 50 percent of the lowest reporting concentration or as otherwise specified by the reference method.
46	What corrective action is taken if the blank is not acceptable? [SM 4020 B-2014 (5)] [15A NCAC 2H .0805 (a) (7) (B)] ANSWER:		SM: Samples analyzed with a contaminated blank must be reprepared and re-analyzed unless concentrations are 10 times those of the blank, concentrations are non-detect, or data user will accept qualified data.
47	Is a calibration blank analyzed prior to sample analysis, after every tenth sample and at the end of the run? [15A NCAC 2H .0805 (a) (7) (H)]		A calibration blank and calibration verification standard shall be analyzed prior to sample analysis, after every tenth sample, and at the end of each sample group, unless otherwise specified by the method, to check for carryover and calibration drift.
48	Is a calibration verification standard (CCV) analyzed prior to sample analysis, after every 10 th sample, and at the end of each sample group? [15A NCAC 2H .0805 (a) (7) (H)] True Value:		
49	What is the acceptance criterion for the CCV? [SM 4020 B-2014 (1) (c)] ANSWER:		Use the same criterion as the back-calculated standard criteria (Question 35). Unless otherwise specified in individual methods, if any recalculated values are not within the method's acceptance criteria—up to twice the MRL \pm 50%; between 3 and 5 times the MRL \pm 20%; or greater than 5 times the MRL \pm 10%—identify the source of any outlier(s) and correct before sample quantitation.

50	What corrective action is taken if the CCV is not acceptable? [SM 4020 B-2014 (1) (c)] [15A NCAC 2H .0805 (a) (7) (B)] ANSWER:		If the CCV is out of control, then take corrective action—including re-analysis of any samples analyzed since the last acceptable CCV.
51	Does the laboratory analyze a laboratory-fortified blank (LFB) with each batch of 20 or fewer samples? [SM 4020 B-2014 (6)] List value(s) and acceptance criterion of standard used. ANSWER:		An LFB is used to evaluate laboratory performance and analyte recovery in a blank matrix. Its concentration should be high enough to be measured precisely, but not high enough to be irrelevant to measured environmental concentrations. The analyst should rotate LFB concentrations to cover different parts of the calibration range. As a minimum, include one LFB with each sample set (batch) or on a 5% basis, whichever is more frequent. (The definition of a batch is typically project-specific.) Process the LFB through all sample preparation and analysis steps. Use an added concentration of at least 10 x MDL, at or below the midpoint of the calibration curve, a method-specified level, or a level specified in a project plan's data quality objectives. LFB may be same source or second source.
52	What is the acceptance criterion for LFB recovery? [SM 4020 B-2014 (6)] ANSWER:		Evaluate the LFB for percent recovery of the added analytes by comparing results to method-specified limits, control charts, or other approved criteria.
53	What corrective action is taken if the LFB recovery is outside established control limits? [15A NCAC 2H .0805 (a) (7) (B)] ANSWER:		If quality control results fall outside established limits or show an analytical problem, the laboratory shall identify the Root Cause of the failure. The problem shall be resolved through corrective action, the corrective action process documented, and any samples involved shall be reanalyzed, if possible. SM states: Establish corrective actions to take if the LFB does not satisfy acceptance criteria.
54	Is a Laboratory Fortified Matrix (LFM)/Laboratory Fortified Matrix Duplicate (LFMD) analyzed with each batch of 20 or fewer samples? [SM 4020 B-2014 (2) (g)]		Laboratory fortified matrix is the same as a matrix spike; that is, a spiked sample. Note: No option to perform an environmental sample duplicate and then spike separately – must perform MS/MSD for this method. SM states: Include at least one LFM/LFMD daily or with each batch of 20 or fewer samples. Prepare LFM from the same source used for LFB.
55	How is the LFM (spike) prepared? [SM 4020 B-2014 (7)] ANSWER:		See Matrix Spike Technical Assistance document for volume and sample dilution requirements. SM states: Prepare LFM from the same reference source used for LFB. Make the addition such that sample background levels do not adversely affect recovery (preferably adjust LFM concentrations if the known sample is more than 5 times the background level). At a minimum, the spike must at least equal the background concentration, unless the method specifies otherwise. For example, if the sample contains the analyte of interest, then add approximately as much analyte to

			the LFM sample as the concentration found in the known sample.
56	<p>What is the acceptance criterion for LFM/LFMD recovery? [SM 4020 B-2014 (7)]</p> <p>ANSWER:</p>		<p>SM states: Evaluate LFM results for percent recovery; if they are not within control limits, then take corrective action to rectify the matrix effect, use another method, use the method of standard addition, or flag the data if reported. See method for specific LFM acceptance criteria until the laboratory develops statistically valid, laboratory-specific performance criteria. If the method does not provide limits, use the calculated preliminary limits from the IDC (4020 B.3). LFM control limits may be wider than for LFB or LCS, and batch acceptance generally is not contingent upon LFM results.</p>
57	<p>What corrective action does the laboratory take if the LFM/LFMD results are outside of established control limits for accuracy? [15A NCAC 2H .0805 (a) (7) (B)]</p> <p>ANSWER:</p>		<p>If quality control results fall outside established limits or show an analytical problem, the laboratory shall identify the Root Cause of the failure. The problem shall be resolved through corrective action, the corrective action process documented, and any samples involved shall be reanalyzed, if possible. Compare to LFB result and other QC. Reanalyze LFM. If it still fails, qualify the spiked sample result.</p>
58	<p>What is the acceptance criterion for LFM/LFMD relative percent difference? [SM 4020 B-2014 (8)]</p> <p>ANSWER:</p>		<p>SM states: When the value of one or both duplicate samples is $\leq 5 \times \text{MRL}$, the laboratory may use the MRL as the control limit for percent recovery, and the duplicate results are not used to measure precision.</p> <p>Bottom line: We are not requiring control charts but will instead accept a system of trend analysis. That is, the lab's monitoring of the trends in the data. 40 CFR part 136.7 (viii) states: Control charts (or other trend analysis of quality control results).</p>
59	<p>What corrective action does the laboratory take if the LFM/LFMD results are outside of established control limits for precision? [15A NCAC 2H .0805 (a) (7) (B)]</p> <p>ANSWER:</p>		<p>If quality control results fall outside established limits or show an analytical problem, the laboratory shall identify the Root Cause of the failure. The problem shall be resolved through corrective action, the corrective action process documented, and any samples involved shall be reanalyzed, if possible.</p>
60	<p>Is the data qualified on the Discharge Monitoring Report (DMR) or client report if Quality Control (QC) requirements are not met? [15A NCAC 2H .0805 (a) (7) (B)]</p>		<p>If the sample cannot be reanalyzed, or if the quality control results continue to fall outside established limits or show an analytical problem, the results shall be qualified as such.</p> <p>If data qualifiers are used to qualify samples not meeting QC requirements, the data may not be useable for the intended purposes. It is the responsibility of the laboratory to provide the client or end-user of the data with sufficient information to determine the usability of the qualified data.</p>

Additional Comments:

Inspector: _____ Date: _____

08/25/2021